Dielectric properties of frozen clay and silt soils

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ABSTRACT

The dielectric properties of a clay and a silt soil have been studied at low water saturations at temperatures between −100°C and −15°C. The frequency range of the study (20 Hz–300 kHz) is lower than most other studies on permafrost. The results show a clear change in phase of the water in the soil at between −65°C and −80°C. The water can be modelled in terms of a thin layer of mobile molecules on the surfaces of soil particles. At low temperatures this layer has a non-Debye-like dielectric dispersion unlike that found for both water or ice. The relaxation frequency of the dispersion and its high activation energy are similar to those found for the quasi-liquid layer on ice particles. At temperatures above the phase transition, the dielectric dispersion is more Debye-like and indicates the presence of low frequency dispersion. We interpret this as due to a thin film of concentrated salt solution lining the soil particle pores giving rise to interfacial polarizations.

Introduction

The electrical properties of permafrost have long been investigated as an aid in understanding geophysical surveys in cold regions. Frozen soil is a complex mixture of mineral particles, ice, water, dissolved salts, and organic material. The relative concentrations of each component changes with temperature, type of soil, and water content. In this paper we discuss the dielectric properties of a natural clay and a natural silt soil at frequencies between 20 Hz and 100 kHz. Most previous efforts have focused on higher frequency measurements using time domain reflectometry (e.g. Delaney and Arcone, 1984), with relevance to VHF radar sounding, or using wave guide methods (e.g. Hoekstra and Doyle, 1971) for microwave remote sensing applications. Few studies have been undertaken on the LF electrical properties of permafrost soils which are relevant in interpreting VLF and DC resistivity surveys (Hoekstra et al., 1975; Sellmann et al., 1989).

Olhoeft (1977, 1978) studied the dielectric properties of permafrost soils in a wide frequency range between 5 mHz and 100 MHz at temperatures between −10°C and −27°C. Frolov (1973) showed results that indicated the specific surface area of the soil was important in determining the electrical properties, and Frolov and Gusev (1973) showed that significant water was unfrozen in clay even at −70°C. Olhoeft (1977) indicated the importance of the unfrozen water in the electrical conductivity of the permafrost, and also the dependence of the electrical behaviour on soil type. Olhoeft (1977) suggested that up to five different dielectric mechanisms were necessary to explain the dielectric response between 5 mHz and 10 GHz. The importance of the unfrozen water and also the interfaces between the different phases of the material were illustrated by the NMR work of Kvlivdze et al. (1980) who showed large differences in the freezing temperature of water in contact with hydrophobic and hydrophilic solids. In systems with only ice–hydrophobic boundaries, mobile water molecules

were detected at $-15°C$ to $-20°C$, while systems with ice–hydrophilic boundaries (such as may be usually expected in permafrost) contained mobile molecules at temperatures of $-50°C$ to $-100°C$. This implies that water in hydrophobic surfaces retains more normal bulk water properties than does water bound on hydrophilic surfaces.

Recently Araki and Maeno (1989) conducted a series of systematic dielectric measurements on natural silt and clay soils over wide ranges of frequency, temperature and water content. They conclude that water molecules existed in the soils in three states that depended on the water content. As the water content of the soils is increased, the water molecules are initially strongly adsorbed on the surfaces of the soil particles, then further water molecules are weakly bound to particles, and finally water is solidified as bulk ice filling the soil pores. Maeno et al. (1992) report the temperature dependence of the dielectric properties found in these measurements; they observe a significant change in dielectric response at a temperature of about $-80°C$, with a change in the activation energy of the dielectric conductivity. In this paper we discuss some of the mechanisms that may be responsible for the different dielectric regimes observed.

Measurements

The dielectric responses of soils of two different types were investigated, namely Fujinomori clay and Tomakomai silt. The particle densities and particle size distributions for the soils are shown in Table 1. There is a striking difference in the concentrations of soluble ions within the two soils (Maeno et al., 1992). Based on the electrical conductivity of water

TABLE 1

Physical properties of the two soils

Particle size range	Fujinomori Clay	Tomakomai Silt
Particle density (Mg m^{-3})	2.61	2.65
2 mm–7.4 μm	15%	39%
7.4–5 μm	61%	32%
<5 μm	24%	29%

soil suspensions around thirty times more impurity ions are available in the Fujinomori clay than in Tomakomai silt.

Sample preparation and the dielectric apparatus have been described earlier (Araki and Maeno, 1989; Maeno et al., 1992). Briefly, the soil samples were prepared by mixing soil with water in bulk then packing the soil into a shallow circular container, the flat bottom of which formed an electrode 80 mm in diameter. The sides of the container were made of insulating plastic; when soil was level with the top of the sides it formed samples about 8 mm thick. Another circular electrode was held in contact with the top of the sample by a weak spring. A guard ring electrode was also used. The water content of each sample was estimated by weighing the sample before and after baking at $120°C$ for 24 hours. The electrodes were then installed in a cryostat bath, regulated by circulating liquid nitrogen. The dielectric measurements discussed here were made between $-100°C$ and $-15°C$.

The samples were measured at high temperature resolution (about $0.5°C$) using a multi-bridge (Wayne Kerr 6425). Maeno et al. (1992) and Araki and Maeno (1989) report other measurement systems with lower temperature resolution but greater sensitivity than that offered by the multibridge. A complete set of high resolution measurements were taken over 24 hours, and the technique is described by Moore et al. (1992). All data were corrected for stray capacitance and residual impedances in the circuit.

Results

We are interested in the effects of water on the dielectric properties of permafrost soils, so firstly we present the response of the material in the absence of water. The dielectric response of dry Tomakomai silt is shown in Figs. 1 and 2 between $-15°C$ and $-100°C$. Araki and Maeno (1989), reported a very similar dielectric response of Fujinomori clay. Notice the large scatter in the data at frequencies below 500 Hz, which is due to the low conductivity of the sample. There is also scatter on the other figures presented here for soils containing water, but generally the noise is severe only at 50 Hz (mains fre-
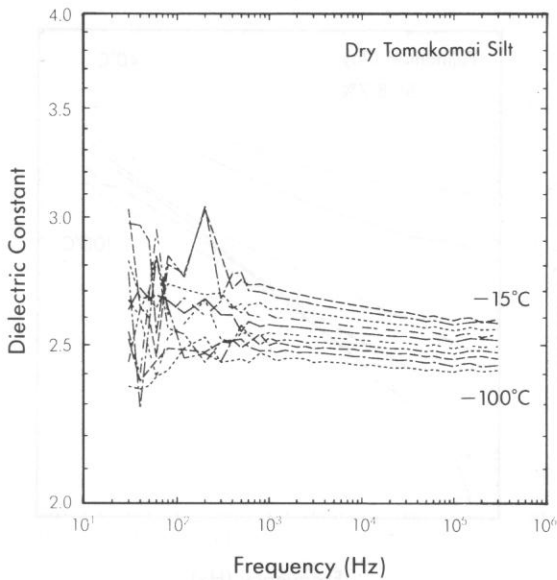
Fig. 1. The dielectric constant of dry Tomakomai silt against frequency. The temperatures (from the top of the diagram) are, $-15, -20, -30, -40, -50, -60, -70, -80, -90$, and $-100°C$.
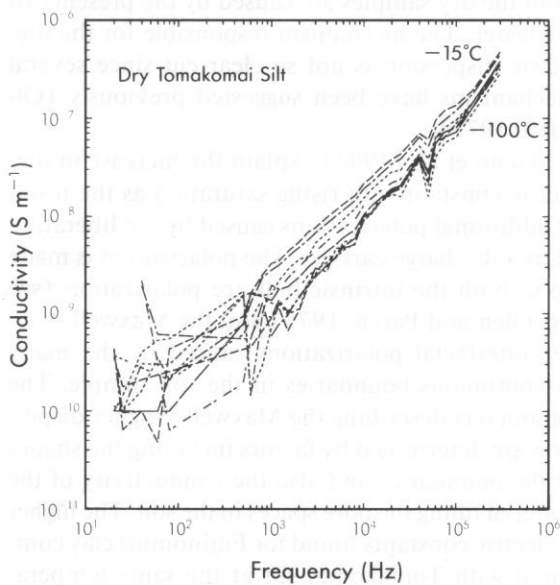
quency) and its first few harmonics. The response of dry Fujinomori clay and dry Tomakomai silt samples do not vary greatly over the frequency and temperature ranges studied here. The conductivities for Tomakomai silt varied from 10^{-10} to 10^{-7} S m^{-1}, and from 10^{-11} to 10^{-7} S m^{-1} for Fujinomori clay. We can regard the conductivities of the dry soils as a small addition to the conductivities caused when water is present in the soil. The small residual dielectric dispersions are not thought to be intrinsic to the soil particles but are the result of small concentrations of charge carriers that remained in the soil after the drying process, or from the moisture that was absorbed from the atmosphere during the experiment.

Figures 3 and 4 show the typical frequency dependencies of dielectric constant and conductivity of Tomakomai silt between $-100°C$ and $-15°C$. The degree of water saturation was 7.7%, where we define degree of saturation as the volume fraction of pore space in the bulk sample that is occupied by water or ice. Figures 5 and 6 give the frequency dependencies of Fujinomori clay at similar saturation of 8.7% but over a smaller range of temperatures. It
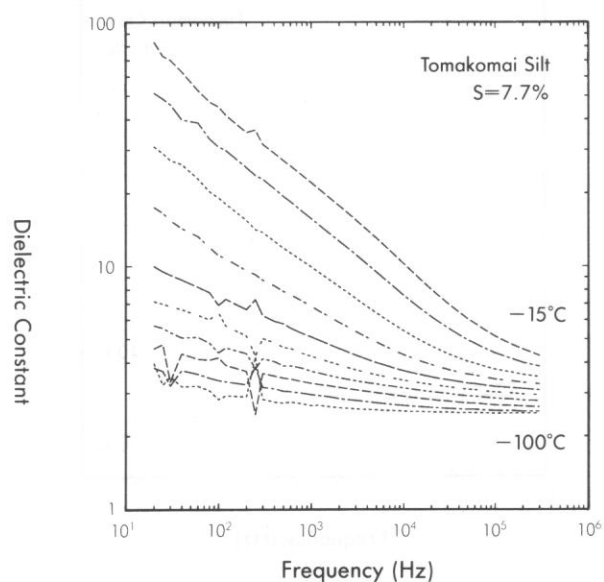

Fig. 2. Conductivity response of dry Tomakomai silt against frequency. The temperatures (from the top of the diagram) are, $-15, -20, -30, -40, -50, -60, -70, -80, -90$, and $-100°C$.

Fig. 3. The dielectric constant of 7.7% saturation Tomakomai silt against frequency. The temperatures (from the top of the diagram) are, $-15, -20, -30, -40, -50, -60, -70, -80, -90$, and $-100°C$.
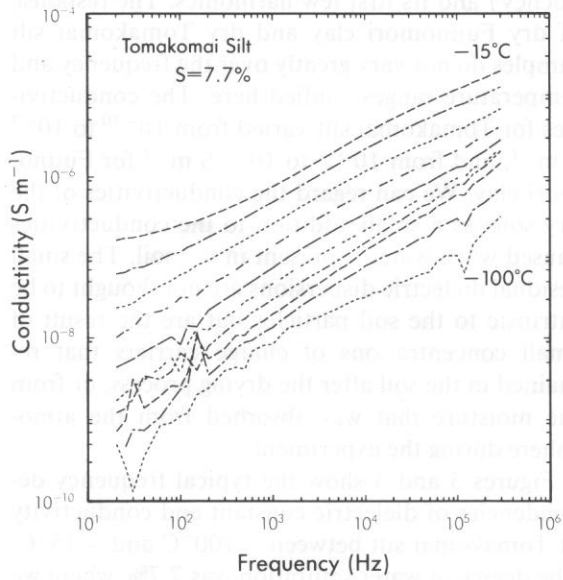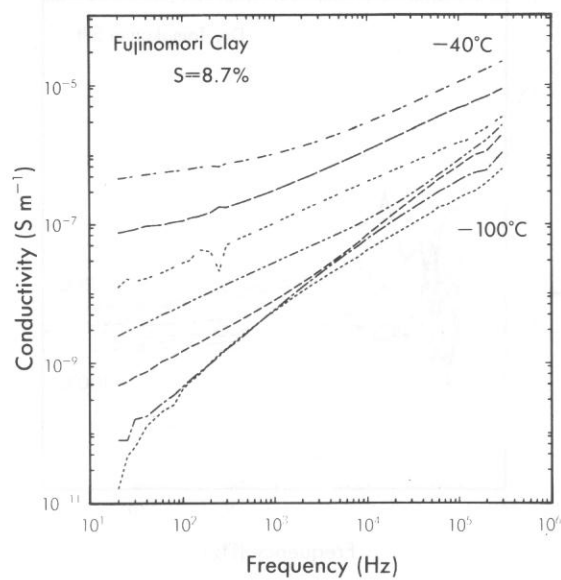
Fig. 4. Conductivity response of 7.7% saturation Tomakomai silt against frequency. The temperatures (from the top of the diagram) are, -15, -20, -30, -40, -50, -60, -70, -80, -90, and $-100°C$.

Fig. 6. Conductivity response of 8.7% saturation Fujinomori clay against frequency. The temperatures (from the top of the diagram) are, -40, -50, -60, -70, -80, -90, and $-100°C$.
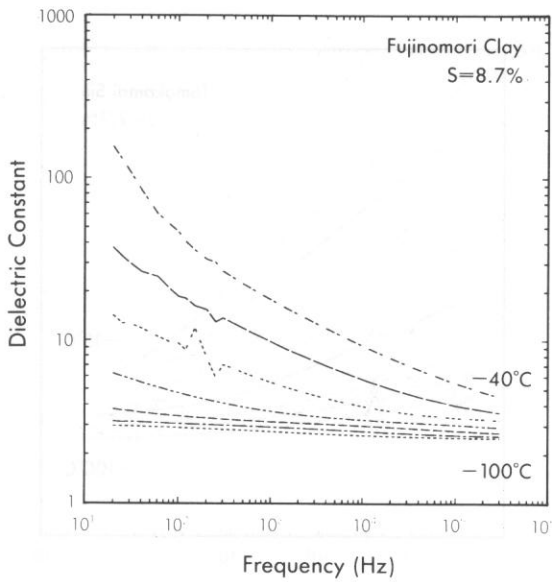
Fig. 5. The dielectric constant of 8.7% saturation Fujinomori clay against frequency. The temperatures (from the top of the diagram) are, -40, -50, -60, -70, -80, -90, and $-100°C$.

is clear that large differences in dielectric response from the dry samples are caused by the presence of the water. The mechanism responsible for the dielectric dispersion is not so clear cut since several mechanisms have been suggested previously (Olhoeft, 1977).

Maeno et al. (1992) explain the increase in dielectric constant with rising saturation as the result of additional polarizations caused by the liberation of mobile charge carriers. The polarization is made up of both the intrinsic bulk ice polarization (see e.g. Glen and Paren, 1975) and the Maxwell-Wagner interfacial polarizations caused by the many discontinuous boundaries in the soil sample. The parameters describing the Maxwell-Wagner dispersion are determined by factors including the shapes of the boundaries and also the conductivity of the material filling the pore spaces in the soil. The higher dielectric constants found for Fujinomori clay compared with Tomakomai silt at the same temperature and similar saturations may be explained by the pores present in clays being smaller and more convoluted than in silt as was observed by Kumai (1988). The even larger differences in conductivity

between the clay and silt suggests a higher pore conductivity caused by the higher concentration of soluble ions in the Fujinomori clay. These observations are consistent with clays being generally strongly ion-exchanging, and the formation of electrical double layers.

Figure 7 shows the 100 kHz conductivity (σ_{100}) plotted against reciprocal temperature for 5 Tomakomai silt samples and one Fujinomori clay sample of saturations between 5 and 11%. The dry Tomakomai silt sample is included for comparison. The general shape of the curves is similar with three distinct sections, a fairly linearly increasing low temperature response, a plateau where σ_{100} is constant, and a high temperature regime, where the rate of increase of σ_{100} seems to increase as the temperature rises. We can model linear parts of Fig. 7 by an Arrhenius type relationship such that

$$\sigma_{100} = VS \exp(-E/kT) \qquad (1)$$

where S is the saturation and V is a constant, k is the Boltzmann constant, T the absolute temperature, and E is the activation energy of conduction.
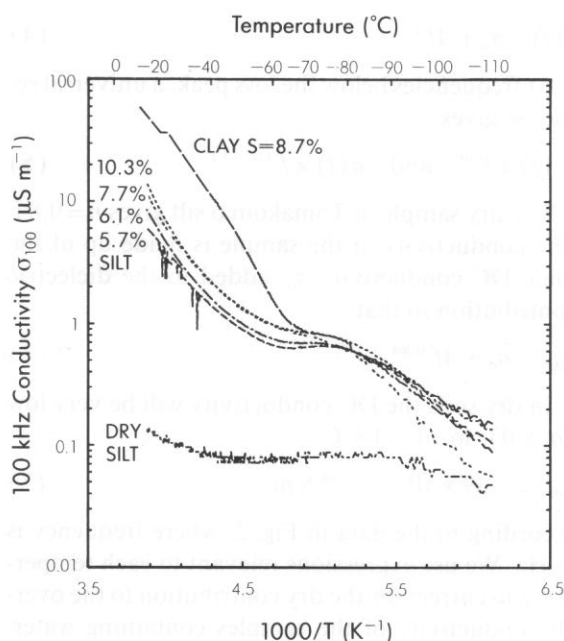

Fig. 7. Plot of 100 kHz conductivity against reciprocal temperature for five Tomakomai silt samples with saturations ranging from dry to 10.3%, and an 8.7% saturation Fujinomori clay sample

From Fig. 7, it can be seen that below about $-80°C$ the activation energies of all the samples (except the dry Tomakomai silt sample) are quite similar, around 0.15 eV (14.5 kJ mol^{-1}). At temperatures warmer than the plateau, the Fujinomori clay sample differs in behaviour to the Tomakomai silt samples. The Fujinomori clay sample plateau has a relatively narrow temperature range between $-65°C$ and $-75°C$, at warmer temperatures the activation energy is about 0.42 eV. The Tomakomai silt samples exhibit a wider conductivity plateau between about $-60°C$ and $-75°C$, the exact limit on the warm side of the plateau is difficult to delimit because of the distinctly concave shape of the conductivity curves. We can estimate an average activation energy for the conductivity of the Tomakomai silt samples between $-60°C$ and $-15°C$ as 0.3 eV.

In contrast to the samples containing moisture, the dry Tomakomai silt sample exhibits a fairly flat response throughout most of the temperature range, the fluctuations at low temperatures are due to measurements limited by the resolution of the multibridge bridge. The increase in conductivity above $-35°C$, corresponds to an activation energy of approximately 0.13 eV (13 kJ mol^{-1}), and probably results from adsorption of water vapour by the sample as it was warmed.

The dielectric spectra of both soils do not show a clear dispersion loss peak at temperatures above $-70°C$. This is in contrast to the results reported by Olhoeft (1977), who found a Debye-like dispersion with a relaxation frequency of around 20 kHz at temperatures of $-20°C$, which he attributes to the Bjerrum defect dispersion found in ice (see e.g. Glen and Paren, 1975). This dispersion is not observed. Instead the behaviour is of the typical "universal" type (Jonscher, 1983) found for a wide variety of different materials ranging from silicon p–n junctions to polyethylene. For temperatures lower than $-70°C$, there is some evidence for a dielectric dispersion. The relaxation frequency of the dispersion can be estimated from the local loss maximum frequency (Fig. 8). The absolute values of loss are very low and hard to measure accurately at these low temperatures, but we estimate at $-100°C$ a relaxation frequency of 500 Hz, and at $-70°C$, the relaxation frequency is greater than 100 kHz, implying an activation energy for relaxation frequency
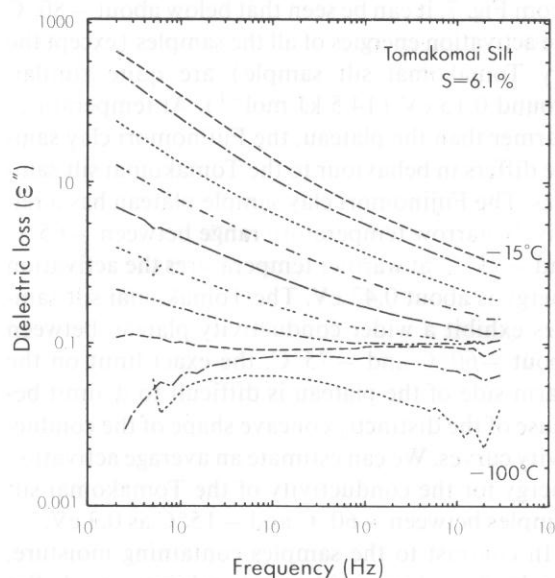
Fig. 8. Logarithmic plot of dielectric loss (ϵ'') corrected for the contribution from the dry Tomakomai silt sample, against frequency for a 6.1% saturation Tomakomai silt sample. The temperatures are (from the top of the diagram) -15, -20, -30, -40, -50, -60, -70, -80, -90, and $-100°C$. Notice the change in response type from the universal behaviour at warm temperatures with near linear variation of log loss with log frequency, to a nearly frequency independent response at $-80°C$, and a loss peak appearing at lower temperatures. The relaxation frequency of the dispersion appears to increase rapidly from about 500 Hz at $-100°C$ to above 300 kHz at $-70°C$.

of about 0.7 eV (68 kJ mol^{-1}). This is similar to activation energies associated with the Debye dispersion in pure ice (Glen and Paren, 1975), although the soil relaxation frequencies are far too high for pure ice to be responsible.

Discussion

The samples exhibit two different types of dielectric response: at temperatures below $-70°C$, a dielectric loss peak is evident, while above $-70°C$ the response shows no relaxation loss peak in the frequency range studied. Although it is desirable to have dielectric data spanning a wider range of frequencies, in particular measurements at higher frequencies, we can use the Jonscher (1983) model of the universal dielectric response to model the ob-

served response. The behaviour shown in Fig. 8 at temperatures greater than $-70°C$ is of decreasing loss as frequency rises, implying that the measurements are on the high frequency side of a loss peak. The low frequency loss is not due to a simple DC conductivity since we observe that ϵ' varies markedly at low frequencies too.

The conductivity of the soils can be considered to be made up of the dry soil conductivity σ_{dry}, added to that due to the conductivity caused by the water in the sample, σ_{sol}. Jonscher's universal type of response at frequencies above the loss peak should show plots of log ϵ'' and $\log(\epsilon' - \epsilon'_\infty)$ linear against $\log f$, where f is the frequency, ϵ' is the real part of the dielectric permittivity, ϵ'' is the imaginary part of the permittivity, and ϵ'_∞ is the limiting high frequency value of ϵ', and

$$\epsilon''(f)/(\epsilon'(f) - \epsilon'_\infty) = \text{constant} = \cot(n\pi/2) \quad (2)$$

where n is a constant, and the frequency response of ϵ'' is given by

$$\epsilon'' \propto f^{(n-1)} \quad (3)$$

so that

$$\sigma(f) = \sigma_o + Af^n \quad (4)$$

At frequencies below the loss peak, a universal response gives

$$\epsilon''(f) \propto f^m \quad \text{and} \quad \sigma(f) \propto f^{(m+1)} \quad (5)$$

The dry sample of Tomakomai silt gives $n = 0.84$. The conductivity of the sample is made up of the ionic DC conductivity σ_o, added to the dielectric contribution so that

$$\sigma_{dry} = \sigma_o + Af^{0.84} \quad (6)$$

In dry soils the DC conductivity will be very low ($\sigma_o \approx 0$), so, at $-15°C$,

$$\sigma_{dry} = 8.39 \times 10^{-12} f^{0.84} \text{ S m}^{-1} \quad (7)$$

according to the data in Fig. 2, where frequency is in Hz. We use expressions relevant to each temperature to correct for the dry contribution to the overall conductivity of the samples containing water. Below $-70°C$, the dielectric response contains a relaxation peak (Fig. 8), and we find a typical value of m, describing the behaviour at frequencies below the loss peak, of about 0.8. The high frequency be-

haviour gives a value for $(1-n)$ of about 0.2, in fairly good agreement with the exponent found for the dry sample over the whole temperature range.

The frequency response of the Tomakomai silt samples above $-70°C$ is of the typical universal character, and can be illustrated by the relationship for the sample of 10.3% saturation at $-15°C$ where

$$\sigma = 5.7 \times 10^{-7} f^{0.25} \text{ S m}^{-1} \qquad (8)$$

The other samples give similar values for the frequency exponent n, of around 0.25, in contrast to the 0.84 found for the dry Tomakomai silt sample, and also for the samples containing water below about $-70°C$.

Jonscher (1983) discusses the physical significance of the exponents $(1-n)$ and m; a classical Debye dielectric dispersion would give an exponent of $m=1$ on the low frequency side of the relaxation frequency. The behaviour of ice is a good approximation to Debye behaviour, with an exponent of about 0.8 at $-90°C$ (Jonscher, 1983) and about 0.95 at higher temperatures. At frequencies above the loss peak a Debye dispersion has $(1-n)=1$, with typical natural Antarctic ice having almost frequency independent conductivity, that is $(1-n) \approx 1$. The Debye behaviour implies a system of independent dipoles, and liquid water is a regarded as a classic example of Debye relaxation with exponents m and $(1-n)=0.96$ (Jonscher, 1983). The closer the exponents are to unity, the less rigid the system, and the individual dipoles more uncorrelated in their transitions from state to state. A change in exponent signifies a phase change in the short range order of the system. In this case, the dramatic change in the value of the exponent $(1-n)$ from about 0.2 at low temperatures to about 0.75 at warmer ones, suggests that the short-range order of the dipoles in the samples decreases as the temperature rises. In the warmer part of the response, the presence of weakly bound water is to be expected (Maeno et al., 1992). This water would be expected to produce an ionic conduction mechanism. The observed data cannot be reconciled with a simple DC conductivity mechanism since ϵ' is also frequency dependent, implying instead a low frequency dispersion. The $(1-n)$ parameter characteristic of the dielectric may then be expected to

increase further to values typical of liquid water, as is observed.

Maeno et al. (1992) show that the dielectric constant of Fujinomori clay varies in a systematic way with water saturation. They account for the behaviour by suggesting that in the range of saturations from 0–5%, water molecules are strongly adsorbed on the surfaces of the soil particles. The adsorption is believed to form a monomolecular layer. The calculated specific surface area of the clay is 9.0 m^2 g^{-1}, implying that the adsorption layer is 1.1 molecules thick at 5% saturation. Between 5 and 10% saturation, the dielectric constant increases rapidly, and is interpreted as due to the development of multi-molecular layers of mobile water molecules or electrical double layers, that can act as charge carriers. Above 10% saturation, the dielectric constant increases less rapidly with increasing saturation, as bulk ice starts to fill the pores. Bulk ice gives a relatively small dielectric contribution and is not detectable unless the water content is rather large (Maeno et al., 1992). This may explain the absence of the 20 kHz dispersion, that Olhoeft (1977) ascribed to bulk ice relaxation, in any of our samples where saturation was very low compared with those of Olhoeft's specimens.

The values of m and $(1-n)$ found for the response at low temperatures indicates a non-Debye-like dispersion, in contrast to the very Debye-like dispersions found in both water and ice. The observation of the high activation energy of the relaxation frequency of the dispersion found in samples with saturations between 5 and 10% at low temperatures, also indicates that the properties of the very thin layers of water/ice are rather different to those of bulk water or ice. Mizuno and Hanafusa (1987) report unusual observations on the surface properties of ice particles, in that they observe NMR correlation times for the surface layer that are much shorter than the value for ice, at about 1/25 the value in water. The NMR correlation time is closely related to the dielectric relaxation time. The correlation time activation energy for their refrozen ice samples was about 0.61 eV. Extrapolating their data from $-50°C$ to below $-70°C$ gives relaxation frequencies of 2.3 kHz at $-100°C$ and 980 kHz at $-70°C$, if the dielectric relaxation time and the NMR correlation time are assumed identical. This

is quite close to the observed relaxation frequency of the dispersion found in the soil samples, which is also the result of thin surface layer of water on the soil particle surfaces. Considering the differences between thin water layers on soil particles and those on ice, the similarity in the relaxation frequency and associated activation energy is remarkable.

The conduction activation energy for the Fujinomori clay sample in the warm part of Fig. 7 is similar to the value (0.52 eV) reported by Hoekstra and Doyle (1971) for Na–montmorillonite between $-15°C$ and $-75°C$ containing roughly three molecular water layers on each clay surface. Weiler and Chaussidon (1968) report activation energies for dielectric loss at 1 kHz of 0.5 eV above $-73°C$ for montmorillonite gels saturated with Li, Na, K, and Cs. The mechanism of conduction may be attributed to conduction via the dissolved impurity ions in the unfrozen water layer. Additionally the hydrophilic nature of the ice/clay interface may also be expected to produce mobile water molecules at low temperatures as observed by Kvlivdze et al. (1978). Sea ice also exhibits an unfrozen fraction at temperatures as low as $-70°C$ (Richardson, 1976). The conduction of concentrated liquid solutions has been studied in relation to acids in polar ice by Wolff and Paren (1984). The activation energy of conduction was well defined and calculated as typically 0.25 eV for HCl, H_2SO_4, and HNO_3 where the proton is the main conducting species. Similar calculations have been done for the behaviour of concentrated NaCl inclusions in ice at low temperatures. The temperature response of the conductivity has an average activation energy of 0.34 eV. The electrical behaviour of salts with lower salt/water/ice eutectic temperature than the NaCl system ($-23°C$) has not been done. The lack of experimental measurements on the electrical conductivity and freezing point concentrations of the salts make such calculations difficult. We simply note that the activation energy of conduction of the Fujinomori clay thus falls well in the range observed by previous authors for clay. The activation energy for the Tomakomai silt samples is rather lower than for the clay; there are much lower soluble impurity concentrations in the silt samples which are also likely to be composed of different salts. The lower activation energy could be a result of the different behaviour of the concentrated salt solution, or alternatively to weaker hydrophilic bonds in the silt compared with the clay.

Conclusions

The dielectric properties of a natural clay and silt soil have been studied. The behaviour can be modelled as water strongly adsorbed onto particle surfaces until a monomolecular layer is formed, and at higher saturations the water molecules form layers of more mobile molecules or electrical double layers. The mobile water molecules appear to change phase between about $-65°C$ and $-80°C$. At cold temperatures the relaxation frequency is intermediate between ice and water and similar to that deduced for the surfaces of ice particles, though the character of the dispersion appears different to both water and ice. At warmer temperatures the mobile water molecules give rise to large dielectric conductivities and polarizations. This is probably a result of the water molecules forming thin quasi-liquid layers on the surfaces of pores in the soils, and generating Maxwell-Wagner interfacial polarizations.

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